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IS : 11678 - 1986

*Indian Standard*

METHOD FOR  
DETERMINATION OF STRENGTH OF  
INDIGO ON DYED TEXTILES

UDC 677·077·4 : 677·047·42 : 667·286·2 : 677·016·8



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**INDIAN STANDARDS INSTITUTION**  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002

# Indian Standard

## METHOD FOR DETERMINATION OF STRENGTH OF INDIGO ON DYED TEXTILES

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# *Indian Standard*

## METHOD FOR DETERMINATION OF STRENGTH OF INDIGO ON DYED TEXTILES

### 0. F O R E W O R D

**0.1** This Indian Standard was adopted by the Indian Standards Institution on 31 March 1986, after the draft finalized by the Dyestuffs Sectional Committee had been approved by the Textile Division Council.

**0.2** The prevalent practice of 'topping' or 'bottoming' indigo dyed materials with other colouring matters has called forth necessity for methods for ascertaining the quantity of indigo present on such dyed materials.

**0.3** The methods prescribed in this standard are based on the extraction of indigo from the dyed textile material by use of pyridine or cresol mixture at the boiling point of the solvent. The accuracy of the method is not affected due to the presence of other dyestuffs in addition to indigo.

**0.4** In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960\*.

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### 1. SCOPE

**1.1** This standard prescribes a method for determining percentage strength of indigo on dyed wool, cotton and linen textile, when present alone or with other dyestuffs.

### 2. PRINCIPLE

**2.1** A suitable quantity of the textile material is extracted with pyridine or cresol mixture at its boiling point. The precipitate obtained on filter is washed successively with hot 50 percent alcohol, hot 2 percent sodium hydroxide solution, hot dilute hydrochloric acid solution ( 1 percent ), hot water, alcohol, and finally alcohol and ether. The precipitate is then dried and weighed. An alternative method is to render the

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\*Rules for rounding off numerical values ( *revised* ).

precipitate of indigo obtained soluble in water by sulphonation with pure concentrated sulphuric acid and titrating with N/50 potassium permanganate solution and then calculating the amount of indigo by the factor: 1 ml of N/50  $\text{KMnO}_4$  is equal to 0.001 47 g of indigotine.

### 3. PREPARATION OF TEST SPECIMEN

**3.1** The mass of the specimen of the cloth or yarn or fibre to be taken shall be such that it gives from 0.03 to 0.10 g of indigo after extraction. This will vary from 3 to 15 g depending upon the depth of indigo present on the textile material. The material shall be loosely packed into the inner tube of the Soxhlet apparatus (see 4.1). If the material to be tested is cloth, it shall be cut into small strips or pieces and then packed into the inner tube in the form of roll.

### 4. APPARATUS

**4.1 Soxhlet Apparatus** — As shown in Fig. 1 which consists of air condenser, in which is placed the inner tube containing the textile material. The inner tube may have the various forms (A, B or C) as shown in Fig. 2. In the apparatus of form A, the hot extract is syphoned intermittently into the distilling flask. With form B, the solvent is directed to the bottom of the tube by means of a long funnel, and then rises through the material overflowing through the side opening near the top. In the form C, it percolates continuously through the material, flowing out by the small hole at the bottom of the tube. The simplest and preferable form is tube C as it gives good results with all cloths and is easy to make. The tube is drawn out at the end, and some glass points are fused on to the outer surface so as to furnish a passage for the vapour of the solvent. A little white wool, cotton wool or crushed quartz is placed at the bottom of the tube, as a filtering and regulating medium.

**4.2 Conical Flask** — 500 ml capacity.

**4.3 A Gooch Crucible** — provided with filter paper or asbestos.

**4.4 Drying Oven** — capable of heating up to  $110 \pm 2^\circ\text{C}$ .

**4.5 Beaker** — 250 ml capacity.

**4.6 Water Bath** — capacity of heating up to 70 to  $80^\circ\text{C}$ .

**4.7 Burette** — graduated to 0.1 ml.

**4.8 Graduated Cylinder** — 100 ml capacity.

**4.9 Titration Flask** — 500 ml capacity.

**4.10 Steam Oven** — for drying.

**4.11 Thermometer** — capable of measuring a temperature up to  $120^\circ\text{C}$ .



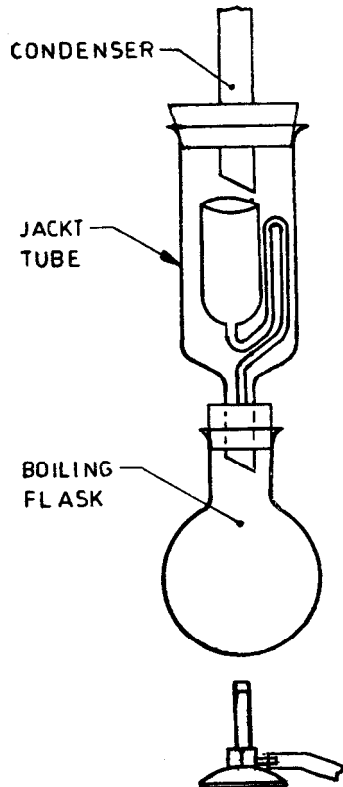
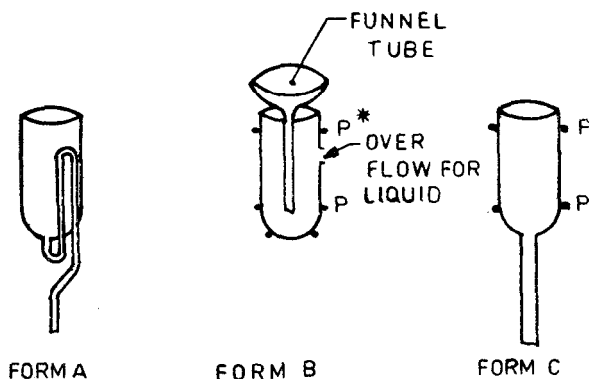


FIG. 1 EXTRACTION APPARATUS



\*P — Glass points fixed on to furnish passage for vapour round the inner tube.

FIG. 2 FORMS OF INNER TUBE

**4.12 Porcelain Beaker** — 250 ml capacity.

**4.13 Burner**

## 5. REAGENTS

**5.0 Quality of Reagents** — Unless otherwise specified, 'pure reagents' and distilled water ( see IS : 1070-1977\* ) shall be employed in the tests.

NOTE — 'Pure reagents' shall mean reagents which do not contain impurities that affect the test results.

**5.1 Commercial Pyridine**

**5.2 Alcohol** — 50 percent (  $v/v$  ).

**5.3 Sodium Hydroxide Solution** — 2 percent (  $m/v$  ).

**5.4 Dilute Hydrochloric Acid** — 1 percent (  $v/v$  ).

**5.5 Ether ( Diethylether )**

**5.6 Pure Concentrated Sulphuric Acid ( AR Grade )**

**5.7 Potassium Permanganate Solution** — N/50.

**5.8 Solvent Mixture** — A mixture of 75 parts of cresol with 25 parts of heavy petroleum spirit ( b p 155° to 170°C ).

\*Specification for water for general laboratory use ( *second revision* ).

**5.9 Sulphuric Acid** — 80 percent ( *m/v* ).

**5.10 Dilute Ammonia** — 10 percent ( *m/v* ).

## 6. PROCEDURE

### 6.1 Estimation of Indigo on Textiles

**6.1.1** Weigh the required quantity of the dyed textile ( *see 3.1* ) accurately after heating it in an air oven at  $105 \pm 3^{\circ}\text{C}$  for 2-4 hours to constant mass and pack it loosely into the inner tube of the Soxhlet apparatus ( *see Fig. 1* ).

**6.1.2** Take 100 ml of commercial pyridine in the boiling flask of the Soxhlet apparatus and heat it over wire-gauze or upon an air bath. Continue the extraction until the extract no longer has a blue colour. This usually requires two hours. The thickest material can be completely extracted in four hours at most.

NOTE — Instead of pyridine, a cresol mixtute ( *see 5.8* ) may also be used for extraction.

**6.1.3** Distil down the extract to about 20 or 30 ml and cool the extraction flask along with contents when the greater part of the indigo separates in well-formed bronzy crystals. To complete the precipitation add to it 100 ml of 50 percent alcohol.

**6.1.4** Heat the contents of the flask to boil for about 10 to 20 minutes and filter through a Gooch crucible provided with filter paper or asbestos into a beaker.

**6.1.5** Wash the precipitate on filter successively with hot (  $50^{\circ}\text{--}60^{\circ}\text{C}$  ) 50 percent alcohol, hot 2 percent sodium hydroxide solution, hot dilute hydrochloric acid solution ( 1 percent ), hot water, alcohol and finally with alcohol and ether.

NOTE — The appearance of the indigo is a guide to its purity. It should form a bronzy crystalline powder testing 100 percent by the tetrasulphonic acid method ( *see IS : 11636-1986\** ). A dull appearance shows the presence of impurities.

**6.1.6** Collect the washed precipitate on the gooch crucible, the bottom of which is covered with a little absetos and dry it for 10 to 20 minutes. Place the crucible in a small beaker containing 15-20 ml of pure concentrated sulphuric acid and heat in water bath at  $70$  to  $80^{\circ}\text{C}$  for about 45 minutes.

**6.1.7** Take the solution in the titrating flask of 500 ml capacity and then make it up to the mark with distilled water. Titrate 100 ml of this

\*Methods for determination of strength of indigo in substance.

solution in 200 ml water against N/50 potassium permanganate solution till end point is obtained ( *see* Note ). Note the volume of N/50 potassium permanganate solution required to reach the end point.

NOTE — The end point is obtained when the solution has a pale yellow or orange colour free from any bluish or greenish tint.

**6.1.8** Repeat the procedure as given in **6.1.7** for remaining 400 ml solution 4 times and find the average of volume of potassium permanganate used.

**6.1.9** Calculate the percentage strength of indigo on the textile by the following formula:

$$S = \frac{0.00147 \times V \times 5 \times 100}{M}$$

where

$S$  = percentage strength of indigo on the textile,

$V$  = average volume of N/50  $\text{KMnO}_4$  used ( *see* **6.1.8** ), and

$M$  = mass of the test specimen ( oven dry basis ) ( *see* **6.1.1** ).

## **7. REPORT**

**7.1** Report the percentage strength of indigo ( **6.1.9** ) present on the textile.

## **8. SAMPLING**

**8.1 Lot** — The quantity of one definite type and quality of textile material dyed essentially under similar conditions delivered to a buyer against one despatch note shall constitute a lot.

**8.2** Sample shall be drawn so as to be representative of the lot. Sample drawn in accordance with the material specifications or as agreed to between the buyer and the seller shall be held to be representative of the lot.